

# Development of a method to quantify perfluorinated compounds and oestrogens present in water by SPE-HPLC-MS/MS

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## Introduction

Perfluorinated compounds and oestrogens are molecules which pollute rivers. Perfluorinated compounds are rejected for the greater part by factories, and oestrogens by human and animals through excretions. These very resistant compounds can be dangerous, even fatal for aquatic organisms and dangerous for the human health even at low concentrations. The commercial stake for CARSO is to be capable of measuring these molecules up to the nano-gram per litre of water. In fact, the authorities need this indication to follow the rejections evolution in rivers. This article shows the various stages of the development of an analytical method allowing the dosage of these molecules. This method uses solid phase extraction (SPE) and high-performance liquid-phase chromatography coupled with tandem mass spectrometry (HPLC-MS / MS).

## Materials and methods

During the development of an analysis method in SPE-HPLC-MS/MS, the detection parameters are determined first. Then, the chromatography conditions are chosen and to finish the SPE conditions are optimized.

The detector is a mass spectrometer equipped with a triple quadrupole analyzer and an electrospray source. The first stage of development consists in injecting directly the molecules, one by one, in the spectrometer with the aim of determining the MRM transitions for each of them. In the same way the MS/MS parameters are optimized to increase the intensity of these transitions. These parameters are: the declustering potential which eliminates the trace of solvent around the molecule during the ionization, the collision energy supplied in the precursory ion that allows it to break up, and the exit potential which guides the fragment ions towards the last quadrupole. After the determination of the mass parameters, the optimization of chromatography can start.

In liquid chromatography, it is necessary to choose a column and mobile phases allowing a separation of molecules, this is achieved by reading the literature and by carrying out many tests. The chosen column is of type C18 end-capped, whereas the used mobile phases are at basic. After several tests a gradient of elution was determined allowing the separation of perfluorinated compounds and oestrogens. The analytical method was established, it is now possible to search the best extraction method to concentrate that compounds at a rate of one nano-gram per liter of water. In SPE, the objective is to obtain a good extraction yield so as to concentrate samples before the analysis. For that purpose, after a step to condition the cartridges, 500mL of sample is loaded in an anion-exchange cartridge. Then, the cartridges are cleaned with water, dried under a stream of nitrogen, and the molecules of interests are eluted with a solvent judiciously chosen. To finish the solvent is evaporated to 250µL. The concentration factor obtained thanks to the SPE is of 2000.

## Conclusion

The developed method is capable of quantifying the perfluorinated compounds and the oestrogens present in water to the state of trace (1ng/L). This allows the authorities to follow the quantities of these compounds rejected in water and to reduce them when they reach the limits fixed by the European Commission. Furthermore, it gives indications on the performances of the water-treatment stations to eliminate these compounds in water.

Figure: Extract of chromatograph shows the separation of three oestrogens.

